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**Title:** Electrochemical corrosion monitoring in low conductive fluid : pilot-scale study on sulfolane corrosion potential

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## **ELECTROCHEMICAL CORROSION MONITORING IN LOW CONDUCTIVE FLUID: PILOT-SCALE STUDY ON SULFOLANE CORROSION POTENTIAL**

keywords: corrosion, sulfolane, aprotic solvent, carbon steel

### **Abstract:**

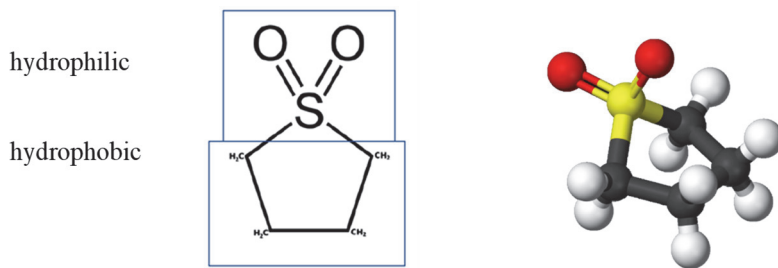
Solvents are a group of chemical compounds that are widespread in the organic chemistry. Taking into account the chemical nature, solvents are divided into the protic and aprotic ones. An attractive alternative to commonly used industrial extractive liquids is an anthropogenic, organosulfur medium – sulfolane. Sulfolane is a five-membered heterocyclic sulfur-organic compound from the group of sulfones ( $\text{ArSO}_2$ ), which contains an apolar hydrocarbon backbone and a polar functional group. It is a selective solvent in the liquid-liquid and liquid-vapour extraction processes used for the removal of close-boiling alkanes from cycloalkanes or for the separation of compounds with different degree of saturation and polarity in the extractive rectification of arenes from non-aromatic

saturated hydrocarbon mixtures. In the standard conditions sulfolane is not an aggressive solvent for steel, but at higher temperature (170-180°C) and oxygen availability, it may be decomposed and subsequently some corrosive (by-)products can be formed. The primary purpose of the presented pilot-case study was to verify the applicability of the industrial, multi-electrochemical technique for reliable detection of the corrosion processes in the low conductive fluids.

## Introduction

A worldwide rise of atmospheric pollution due to the expansion of industrial/agricultural areas and urban settlements is a peculiar 'landmark' of the modern civilization. The global ecosystem is confronted with volatile organic compounds (VOCs) as well as inorganic odorous compounds (VICs) that pose hazards to health of human beings and plants vegetation forming a significant part of indoors/outdoors pollution [1]. Pollutants emitted from liquids originate from anthropogenic activity and/or the biogenic emissions of reactive hydrocarbon derivatives formed in the result of the natural transformations. The odor-producing mixtures of sulfur-based pollutants compose a relevant group of contaminants with increasingly detrimental impact on human health in the long-term exposure [2]. The individual sulfur derivatives exert some irritant and toxic effects being classified as a potential mutagenic and carcinogenic risk factors. In fact, a wide range of environmental contaminants was rationally designed and engineered for industrially-specific targets. On the other hand, impurities such as hydrogen sulfide ( $H_2S$ ) or carbon dioxide ( $CO_2$ ) are constantly present in the natural gases, the removal of which, is a part of 'sour' gas refinement process. In particular, the process of dispatching the acidic gases, composed of  $H_2S/CO_2$  mixture, to 'soften' the natural/industrial (off)gases is called gas treatment [3]. The increasing public awareness of necessity for environmental protection is the main driving force for the increasingly stringent regulations governing release of hazardous pollutants and reduced sulfur compounds (RSCs) as well. Hence, the environmental legislations are constantly pushing industry for developing/optimizing of the cost-effective 'green' manufacturing technologies that impose less burden on the ecosystem [4]. In consequence, the removal of sulfur-containing compounds (e.g.  $H_2S$ , COS, mercaptans, organic sulfides) from unprocessed natural gases can be conducted in the liquid-liquid extraction process, where industrial solvents are applied in a 'closed' loop, recovered and regenerated on-site [5]. Regarding this context, the questions about the 'green' solvents and manufacturing procedures for the liquid-liquid extraction are pretty important. In fact, a range of solvents can be employed in the extraction process used in oil refining including (di/tri/tetra)ethylene glycol (D/T/TT/EG), diglycol amine (DGA), n-methyl pyrrolidone (NMP), dimethylsulphoxide (DMSO), dimethylformamide (DMF), morpholine and carbonate derivatives. An

attractive alternative to commonly used industrial extractive liquids is sulfolane ( $C_4H_8SO_2$ ), an anthropogenic organosulfur medium that is widely distributed in the industry due to its physicochemical properties. Although the spelling ‘sulfolane’ may be astonishing for British English users, this name has been widely accepted as the generic name for hydrogenated sulfones of butadiene [6]. On the other hand, sulfolane is also known under a variety of synonyms/numbers including thiolane 1,1-dioxide (IUPAC), 2,3,4,5-tetrahydrothiophene-1,1-dioxide (systematic), thiocyclopentane-1,1-dioxide, (cyclo) tetramethylene sulphone, dihydrobutadiene sulphone, sulphoxaline, 126-33-0 (CAS) or 204-783-1 (EINECS), respectively [7]. Formally, sulfolane belongs to a group of cyclic sulfone, containing a four-membered carbon ring and sulfonyl functional group ( $R_2SO_2$ ) with a sulfur atom double-bonded to two oxygen atoms as shown in Figure 1.



**Fig. 1.** Structure of sulfolane with (a) hydrophilic and hydrophobic parts and (b) 3D geometry.

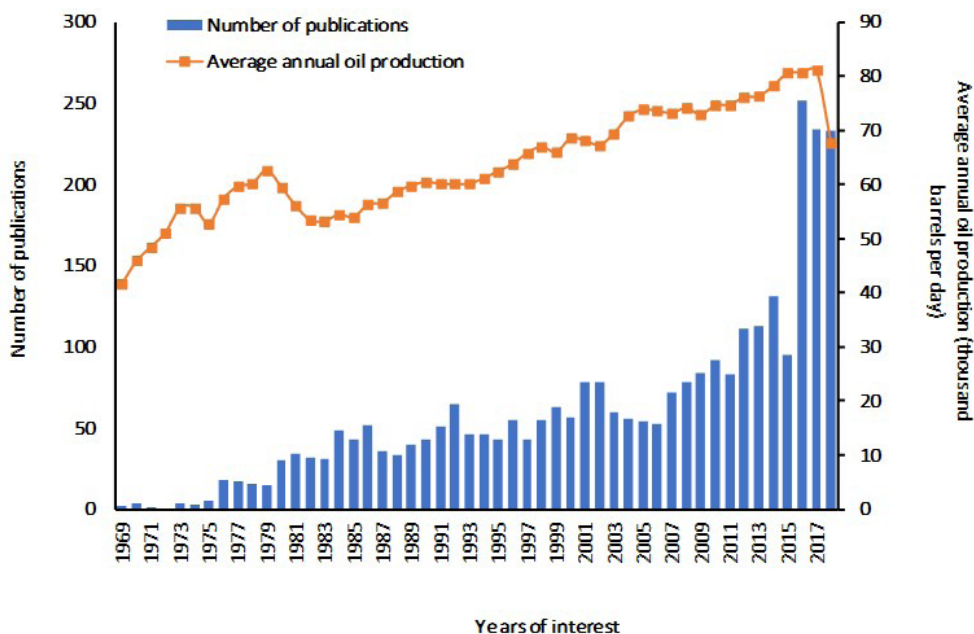
Source: [Own study].

As a matter of fact, sulfolane was the first time described in the chemical literature in 1916; however the study for commercial application of sulfolane were initiated in the early 1940s [8]. Due to its satisfactory selectivity, low boiling temperature and its capacity for dissolving large quantities of aromatics sulfolane is the preferred solvent in the liquid-liquid and liquid-vapour extraction processes for the separation of compounds with various degree of saturation and polarity in the extractive rectification of BTX (benzene, toluene, xylene) mixture from the non-aromatic saturated hydrocarbons [9]. The unique combination of physical and chemical properties (inertness and stability) makes it possible to apply sulfolane not only as a ‘sour gas sweetener’, but also as a biomedical reagent (g.e., sulfolane-induced hypothermia) [10].

It begs the question of whether sulfolane is an appealing liquid to the scientific community? In fact, we found some highly interesting regularity while exploring the commercially available Reaxys database [11]. Oddly enough, a visible tendency of waxing and waning interest in sulfolane during the space of the

last fifty years is observed in Figure 2 as indicated the analysis carried out on the data stored in the Reaxys database. Basically, 1644 hits were identified with the word 'sulfolane' in the title or abstract of the paper published in the range of the last fifty years (from 1969 to 2018) – most of them describe sulfolane physicochemical properties (approximately 43%).

It is natural to ask the question what actually induced a growing interests in sulfolane that fades after some years? Not surprisingly, the waves of interests correlate quite well with the general trends observed on the commercial market of oil production as indicated in Figure 2.



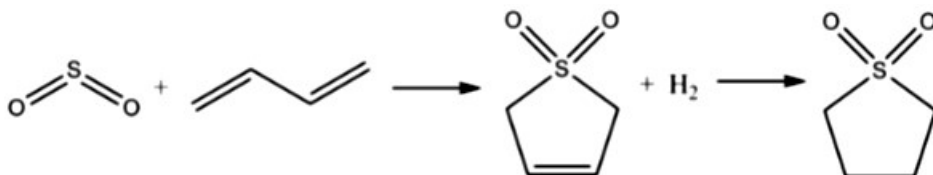
**Fig. 2.** Trends in the average annual crude oil production and interests in sulfolane during the space of last fifty years. Plot of the number of papers, where sulfolane was searched in paper title or abstract and average annual oil production (thousand barrels/day) based on Reaxys and EIA data [11,12].

Source: [Own study].

Basically, the constant growth in the annual oil production is observed throughout the last five decades that is directly related with the increased global oil consumption. It generally means that some 'magic extractors' are urgently needed in the petrol-related industry for the dearomatization of petroleum fractions and (pre)treatment of natural gases, respectively. In other words, the indicated 'wavy' trends in the sulfolane-related scientific activity is an illustration of the partial correlations with the annual oil production. It is noticeable

that even some 'plateau' in the number of published papers superimpose with the periods of the relative annual oil production as observed between 1984 and 1987 or at the beginning of new millennium as well. It is obvious that economic aspects of the aromatic extraction in the refining processes used in the oil industry put pressure on the scientific community.

Originally, the process of sulfolane synthesis was composed of three basic stages involving the hydrogenation of 3-sulfolene (**c**) obtained as a product of sulfur dioxide (**a**) and butadiene (**b**) reaction as illustrated in Scheme 1.



**Sch. 1.** Industrial process of sulfolane synthesis.

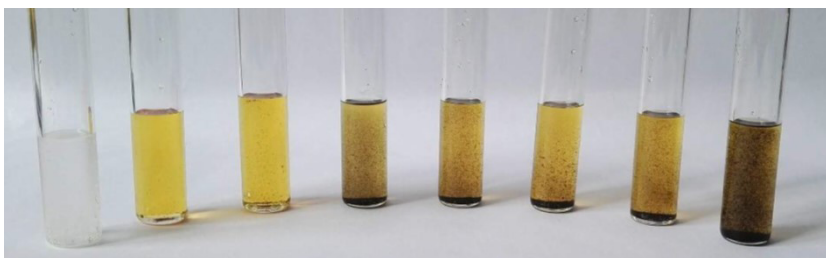
Source: [Own study].

Regarding the environmental constraints the introduction of the eco-friendly catalytic protocols was promoted in the contemporary methods for sulfolane preparation with the operational simplicity, rapid reaction rates and effective formation of the easily handled reagents, respectively [13]. Recyclable pollution-free catalysts reused in the mild reaction conditions with halogen-free solvents and 'green' oxidants are attractive alternative for the large scale industrial sulfolane production estimated at 18,000 – 36,000 tons per year [14]. Theoretically, large-scale industrial usage of sulfolane is disposal/leakage-free due to its on-site recovery and regeneration; however in practise some unpredicted/accidental spills as well as leaks from extraction units in refineries or gas plants were reported worldwide resulting in the soil and groundwater contamination, therefore the environmental fate of the compound is being extensively studied [15]. In this context naturally appears the question about the major reasons of the accidental leaks and spills of sulfolane. Is it important to monitor the sulfolane-induced corrosion of the industrial systems since pure sulfolane under standard operating conditions is considered to be a stable compound and non-aggressive to steel? Unfortunately, the answer to question is positive, because sulfolane-containing systems, if contaminated by traces of oxygen and at typical process conditions (170-180°C), may lead to sulfolane decomposition and formation of corrosive (by-)products with generation of SO<sub>2</sub> and formation of corrosive H<sub>2</sub>SO<sub>3</sub> according to the following equation:





Hence, the corrosion of steel can be quite rapid, causing severe damage to industrial installations, therefore sulfolane should be stored under a nitrogen blanket and out of contact with oxygen and strong oxidizing agents such as chlorates, nitrates and peroxides, that can result in its decompositions. Moreover, the presence of impurities such as oxygen and/or chlorides or water can accelerate sulfolane 'breakdown' process as well. In fact, some general correlations between oxygen/chloride and water concentrations are described, where sulfolane degradation and production of acidic corrodents lead to enhanced sulfolane corrosivity [16,17]. For instance, oxygen-degraded sulfolane is characterised by a lower pH with lower extractive power, a higher acid number and a darker color compared to pure sulfolane as depicted in Figure 3.



**Fig. 3.** Samples of sulfolane, from the left: pure sulfolane, sulfolane after the experiment at 95 °C, sulfolane with water 2%, 3%, 4%, 5%, 6%, 6% with oxygen and 6% with chlorides.

Source: [Own study].

It should be emphasised that there is 'a gap on the path from data to knowledge' in the detailed and quantitative assessment of the individual impurities on the sulfolane-induced corrosion of carbon and alloyed steels.

The main purpose of the previous and current pilot-case studies was to verify the applicability of the industrial, multi-electrochemical technology for reliable detection of corrosion processes in the fluids with low conductivity. Several aspects of the corrosion measurement were taken into account, including the influence of process parameters (temperature as well as the impact of impurities, e.g. water, oxygen and chlorides) on the corrosion of carbon steel in the pure sulfolane. In our studies we used the SmartCet® by Honeywell whose operation is based on Low Frequency Impedance (LFI), Harmonic Distortion Analysis (HDA) and Electrochemical Noise (ECN). The industrial, wired transmitter, model CET5500 was used conjugated with the HART (Highway Addressable Remote Transducer) system. The set of the flat coupon electrodes made of AISI 1010 carbon steel was used (dimensions: 89 × 20 × 2 mm) in the study. Moreover, a dedicated testing vessel was designed and constructed. The electrodes were supported on a special, grooved glass frame to ensure their stability

during the experiment and to maintain contact with the solvent. Several aspects of the corrosion measurement of general and localized corrosion modes were scrutinized extensively. The incipient attempts to quantify impact of process parameters (temperature) and impurities (e.g. water, oxygen and chlorides) on carbon steel corrosion in the pure sulfolane provided meaningful data [18]. Moreover, the oxygen ingress to the pure sulfolane showed minimal impact on the corrosion rates but engendered enhanced localized corrosion activity. As we observed, chlorides enhanced both general and localized corrosion, respectively. The obtained findings for carbon steel demonstrate the applicability of multi-technique electrochemical monitoring systems for rapid and accurate detection of sulfolane corrosion [17,18].

## Materials and Methods

Pure sulfolane is not considered to be a substance causing or accelerating corrosion and destruction of steel. The corrosion of steel associated with the use of sulfolane is the result of the by-products of the decomposition of sulfolane. The main reason for this type of corrosion is the presence of impurities at individual extraction stages of the sulfolane usage, which accelerate decomposition processes. In particular, oxygen or chlorides in contact with sulfolane cause its degradation. Sulfolan is thermally stable up to about 220 °C when it begins to break down into sulfur dioxide and a polymeric material. Oxygen is present in the extraction by air penetrating into the vacuum processes or by the air dissolved in the hydrocarbon feed. Direct reaction of sulfolane with oxygen creates sulfuric and organic acids as well as aldehydes and ketones. Sulfolane degraded by oxygen has a lower pH, a higher acid number and a darker color than pure sulfolane (Figure 3). In addition, under oxygenating conditions, it tends to reduce the efficiency of aromatic compound extraction in the aromatic extraction process [19].

The main purpose of the described pilot-scale study conducted on carbon steel AISI1010 was preliminary analysis of selected factors that could potentially affect the low-conductivity corrosion rate. Monitoring of specific parameters allows to determine trends, similarities and differences between general and local corrosion mechanisms. Laboratory tests regarding the impact of process parameters (temperature) and pollutants (oxygen and chloride) on the corrosion of the carbon steel in pure sulfolane provided important data and clues that may find the practical application in the large-scale industrial processes. For the purpose of the proposed investigations a dedicated testing vessel was designed and constructed as well. Electrodes were supported on a special, grooved glass frame to ensure their stability during the experiment and maintain contact with the solvent. Electrodes were separated by additional glass spacers. Electrodes were immersed in a round bottom, thick-walled reactor vessel of 500 cm<sup>3</sup> capacity, made of heat-resistant glass. The test vessel was also equipped with



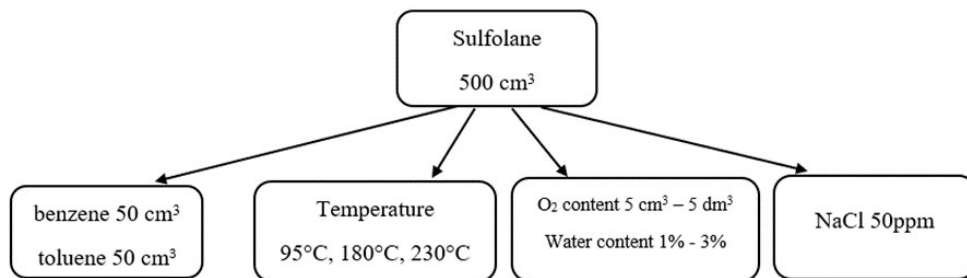
mercury thermometer and a reflux condenser with a moisture adsorber filled with  $\text{CaCl}_2$ . The overall corrosion-test setup is shown in Figure 4. To limit the contact of liquid with air, special silicone-type grease was used on all glass connections. For additional protection against the air ingress, each experiment was conducted under inert gas (99.995% Argon) blanket. The test vessel was placed in a heating bowl and heated to the appropriate temperature. During experiment (c.a. 96h) sulfolane solution was continuously stirred (c.a. 1000 rmp, 1 cm magnetic stirrer) to simulate flowing conditions.



**Fig. 4.** The laboratory-scale setup for sulfolane corrosivity evaluation.

Source: [Own study].

All measurement results were recorded using the industrial electrochemical monitoring system – SmartCET, which uses low frequency impedance (LFI), harmonic distortion analysis (HDA) and electrochemical noise (ECN). Each parameter, e.g. general corrosion rate, local corrosion potential (Pitting factor), current Stern-Geary coefficient – B value and Corrosion Mechanism Index (CMI) were measured and recorded at 60 second intervals using the appropriate data recording system. The brief description of measured parameters is given in Table 1. The experiment time was set to 96 hours on the non-standard electrodes made of carbon steel AISI1010. A series of corrosion test were carried out, maintaining the process temperature in the range of 95°C, 180-190°C and 230-240°C with the pure sulfolane [17,18]. During the experiments with the addition of oxygen, an air was introduced under the surface of the sulfolane solution in the appropriate quantities within 3 days. The influence of chlorides (50 ppm) on the steel corrosion was examined as well. The detailed description of each of the conducted experiments is beyond the scope of this paper; therefore we focused on the introduction of the selected water case study.



**Sch. 2.** Experimental design diagram.

Source: [Own study].

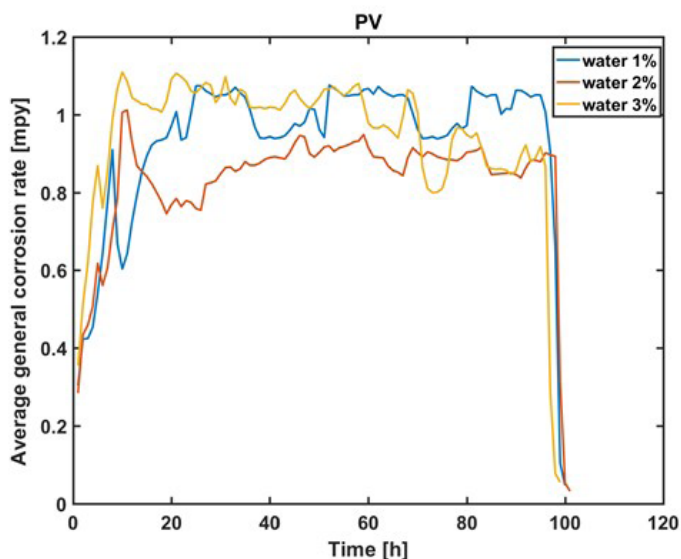
**Tab. 1.** Description of recorded corrosion parameters

Parameter	Definition
General corrosion rate (PV)	Measurement of the real part of the Low Frequency Impedance (LFI) of the working electrode. SmartCET uses Linear Polarization Resistance (LPR) technique to calculate the General Corrosion Rate, that is usually the prime variable of interest, because it reflects the overall rate of metallic corrosion. Corrosion may be directly related to operational parameters, e.g., temperatures, flow, chemical composition.
Pitting Factor (PF)	Ratio of the depth of the deepest pit (point or small area, that takes the form of cavities) resulting from corrosion divided by the average penetration as calculated from weight loss. It is a measure of the overall stability of the corrosion process obtained from a measurement of the intrinsic current noise of the working electrode and comparing this measurement to the general corrosion obtained from the LPR measurement.
Dynamic B value	Corrosion constant also known as the 'Stern-Geary constant'. It is an essential part of the corrosion rate calculation being directly proportional to the corrosion rate value. It represents a correction factor constant determined by the mechanism/kinetics of the corrosion process. In a dynamic process the B value is not constant. The knowledge of the B value enables to refine the LPR-generated corrosion rate estimation, since the uncertainty regarding the standard (default) B value is removed. The B value is directly related to the mechanistic properties of the component anodic and cathodic corrosion processes.
Corrosion mechanism indicator (CMI)	The CMI is a qualitative indicator of a surface film presence. If there is no film and only corrosion is present, the CMI will have an intermediate value. Inorganic scale, or thick passive oxide films with little or no conductivity, will show a low CMI value.

Source: [20].

## Results and Discussion

All experiments were monitored using the Honeywell CET5500 Corrosion Transmitter at selected intervals (60 seconds) and recorded using a computer system for 96 hours. To visualize and interpret the results obtained from the experiments, two software scripts were implemented in the MATLAB environment for downloading and analyzing data from a text file, where a data binning (or bucketing) as a data pre-processing was used to show more accurately the trends of measured values – the average value was calculated from each interval. The implemented software scripts and operational configuration of the laboratory-scale apparatus were applied to monitor the water content (1%, 2% and 3%) and corrosion parameters such as general corrosion rate (PV – primary variable) expressed in mils per year (mpy), localized corrosion potential (dimensionless Pitting Factor), actual Stern-Geary coefficient – B value (mV) and a capacitance-related Corrosion Mechanism Indicator (dimensionless CMI). It should be emphasised that pure sulfolane used for experiments contained less than 0.2% vol. of water; however it was interesting to analyse the impact of water content on the corrosion processes on the carbon steel (AISI1010). Under the extreme of some measurements (temperature  $>230^{\circ}\text{C}$ ), it is obvious that the water would evaporate when the temperature exceeds  $100^{\circ}\text{C}$ , therefore we decided to conduct our experiment at a temperature not exceeding  $95^{\circ}\text{C}$ .

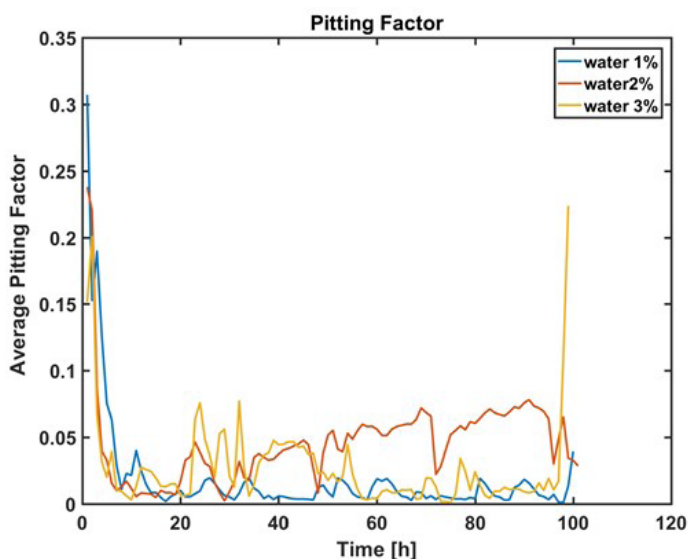


**Fig. 5.** General corrosion rate measured in sulfolane solutions with the addition of 1%, 2% and 3% vol. water at  $95^{\circ}\text{C}$ .

Source: [Own study].

Surprisingly, the general corrosion rate does not increase with the amount of water in sulfolane solution; however some general trends were easily observed. It is evident that, in the first 15 hours of each experiment the average PV parameter is increased and subsequently stabilized with the fluctuations in the range of approximately 0.2mpy as illustrated in Figure 5. We confirmed some general correlations between water concentration and sulfolane corrosivity that were reported in the literature, where water above concentration 3% accelerate sulfolane degradation and production of acidic corrodents [16,17].

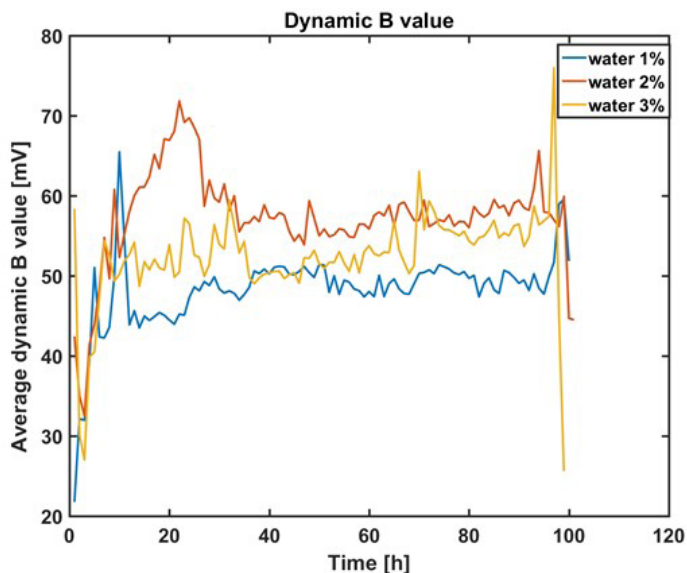
For all experiments with water-contained sulfolane the localized corrosion potential, expressed by Pitting Factor, was at very low level ( $<0.1$ ) indicating domination of general corrosion mechanism as depicted in Figure 6.



**Fig. 6.** Localized corrosion potential measured in sulfolane solutions with the addition of 1%, 2% and 3% vol. water at 95°C.

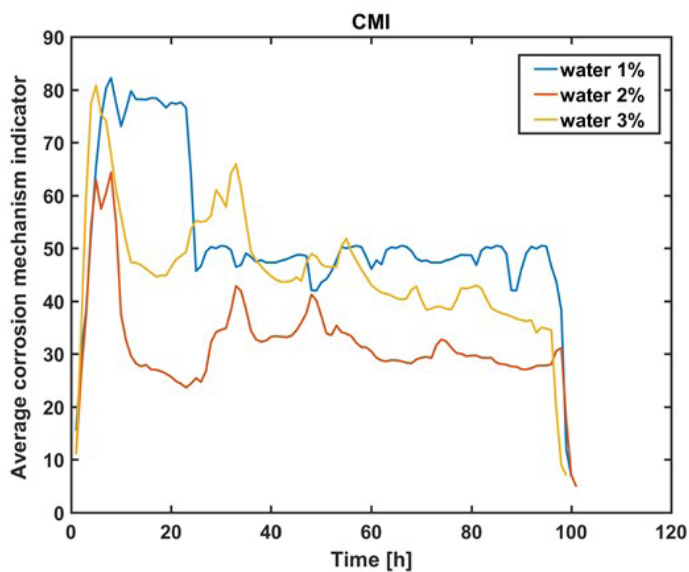
Source: [Own study].

The B value (see Figure 7) is quite stable during the experiment irrespective of water content; however the presence of water at concentration of about 1%vol, at 95°C, generated the lowest level of corrosion. After the initial fluctuations the CMI parameter for the experiments with water addition did not exceed 50 units (see Figure 8), which means that no surface layer was formed during the measurement.



**Fig. 7.** Dynamic B value measured in sulfolane solutions with the addition of 1%, 2% and 3% vol. water at 95°C.

Source: [Own study].



**Fig. 8.** Corrosion mechanism indicator measured in sulfolane solutions with the addition of 1%, 2% and 3% vol. water at 95°C.

Source: [Own study].

An interesting observation was made regarding sulfolane color during the experiments in sulfolane-water system. The more water in the solution the more black sediment was observed as illustrated in Figure 3. It seems a bit surprising since all experiments were performed at about 95°C, that is far below sulfolane decomposition temperature (200-230°C). Apparently, presence of water accelerates decomposition reactions, even at relatively low temperatures, causing more suspended solids, but also increasing the corrosion level.

## Conclusions

A relevant subgroup of water and soil contaminants is composed of the sulfur-based derivatives and their metabolites, sometimes called as ‘old devils of green chemistry’ due to their detrimental impact on human health following long-term exposure. In over-polluted world contemporary chemistry plays a key role in the design of the sustainable, atom-economical and operationally simple procedures to produce eco-friendly (by-)products; however some industrially-engineered solvents are invariably regarded as environmentally unfavorable. On the other hand, the demand for environmental protection is the major driving force for the imposition of the increasingly stringent regulations governing the emission of hazardous pollutants and reduced sulfur compounds. The growing public awareness of necessity for reducing the burden of ecosystem is constantly pushing industry to develop and/or optimize the manufacturing technologies. In fact, the eradication of sulfur-based compounds can be performed in the liquid-liquid extraction process, therefore ‘green’ solvents and cost-effective large-scale procedures are highly desirable. An attractive alternative to commonly used industrial extractive liquids is sulfolane – a versatile dipolar aprotic solvent. Due to its ‘unique’ physicochemical properties as well as the potential of sulfolane to cause equipment corrosion and subsequent spills it can be both regarded as ‘magic extractor’ or ‘bad actor’ [18]. The primary objective of the presented case study was to verify applicability of industrial, multi-electrochemical technique for reliable detection of corrosion in low conductive process fluids. Several aspects of corrosion measurement of general and localized corrosion modes were investigated using the dedicated testing vessel that was designed and constructed. A number of experiments to evaluate the impact of different process parameters on the corrosion behavior of AISI 1010 carbon steel in sulfolane were carried out. In summary, a noticeable impact of water (even at low temperature) on sulfolane corrosivity was observed, where the increase of water concentration accelerates sulfolane degradation as indicated by elevated corrosion rate and increase of suspended dark deposits. It should be highlighted that the described case study should be enhanced including a wider range of solution conditions and the corresponding electrode surface analysis, respectively.



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